

(19)



JAPANESE PATENT OFFICE

PATENT ABSTRACTS OF JAPAN

(11) Publication number: 09110412 A

(43) Date of publication of application: 28 . 04 . 97

(51) Int. Cl

C01B 33/12
B01J 19/00

(21) Application number: 07290386

(71) Applicant: SUMITOMO SITIX CORP

(22) Date of filing: 11 . 10 . 95

(72) Inventor: OKAMOTO NOBORU

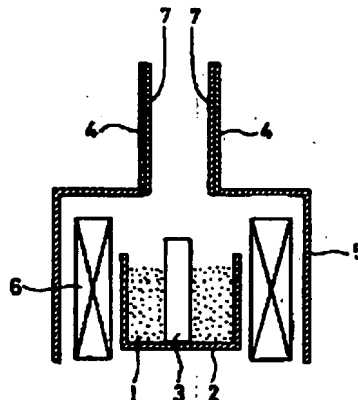
(54) PRODUCTION OF SILICON OXIDE

(57) Abstract:

PROBLEM TO BE SOLVED: To produce silicon oxide suitable for use as an evaporating material when silicon oxide is vapor-deposited on a plastic film by irradiation with electron beams.

SOLUTION: Stock silicon 1 is evaporated by heating with a heater 6 and silicon oxide 7 is deposited on the surface of each substrate 4 made of a metal having a roughened surface structure. A heating element or a heat conductor 3 which is efficiently heated with the heater 6 is set in the stock silicon. The surface of the deposited silicon oxide 7 is made flat and the silicon oxide 7 does not crush at the time of peeling from the substrate 4.

COPYRIGHT: (C)1997 JPO



* NOTICES *

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacture approach of the oxidation silicon used in case oxidation silicon is vapor-deposited on the front face of the plastic film used for a food packaging material etc.

[0002]

[Description of the Prior Art] In recent years, in each field, such as food, medical supplies, an electronic ingredient, and optical-related, since the plastic film which covered the metallic oxide is excellent in transparency, thermal resistance, and gas barrier nature, it attracts attention as wrapping or a gas shielding material, and is developing the need. Although two, an aluminum system and a silicon system, are mainly used as the metallic oxide, an oxidation aluminum covering film has a problem in retort-proof nature. Moreover, the fault that it cannot be used for a microwave oven, and contents cannot be squarely faced since it is opaque although the film which covered metal aluminum excels what used these metallic oxides in gas barrier property exists. To these, the oxidization silicon covering film had both gas barrier property retort-proof nature transparency a sex corresponding to a microwave oven, etc., and has obtained the comprehensive, present highest evaluation.

[0003] There are a spatter and vacuum deposition as an approach of covering oxidation silicon to up to a film. Since the membrane formation rate is slow as compared with vacuum deposition, a spatter is not suitable for fields, such as wrapping of which low cost is required. For this reason, in fields, such as wrapping, vacuum deposition is used abundantly.

[0004] In a vacuum, the oxidation silicon which is a vacuum evaporatio no ingredient is directly heated by resistance heating, induction heating, etc., and the film which vapor-deposited oxidation silicon evaporates it, and is manufactured by making the steam adhere to a film. In this manufacture, in order to prevent the phenomenon (splash) in which oxidation silicon disperses at the time of heating, the measures which make a vacuum evaporatio no ingredient massive are taken.

[0005] Moreover, in recent years, the vacuum evaporatio no which used EB (electron) as a heating means is also increasing. High energy can be centralized on the narrow range and use of EB can also evaporate a metallic oxide with low vapor pressure comparatively easily. However, if the exposure of EB is not carried out to homogeneity but high energy is given partially, the oxygen in a metallic oxide will decompose and the pressure in a system will be raised. For this reason, evaporation thickness becomes an ununiformity or adhesion with a film becomes inadequate. Then, it is necessary to irradiate EB at homogeneity at a vacuum evaporatio no ingredient.

[0006]

[Problem(s) to be Solved by the Invention] In order to irradiate EB at homogeneity at a vacuum evaporatio no ingredient, it is required for the front face of a vacuum evaporatio no ingredient to give surface smoothness. However, because of the condition of having broken, it is very difficult to irradiate EB at homogeneity, and since the massive oxidation silicon currently conventionally used in order to prevent a splash has a break in an ingredient, it cannot irradiate EB continuously.

[0007] There is a cause of the problem that massive oxidation silicon breaks, at the time of manufacture of the oxidation silicon. The massive oxidation silicon used for manufacture of an oxidation silicon vacuum evaporation film as a vacuum evaporation ingredient is conventionally filled up with powdered raw material silicon in a container, carries out heating evaporation of this under a vacuum, and is manufactured by making the front face of the base which consists of a quartz vapor-deposit. However, since the adhesion of a quartz and oxidation silicon is strong, when making the oxidation silicon vapor-deposited on the surface of the quartz exfoliate from a quartz, the oxidation silicon will break.

[0008] On the other hand, the oxidation silicon which metals, such as an SUS plate, had adhesion weaker than the quartz with oxidation silicon, and was vapor-deposited on the front face can be made to exfoliate with the original form. However, when a front face becomes concave convex voice and it uses as a vacuum evaporation ingredient of a metallic-oxide vacuum evaporation film, the homogeneity exposure of EB is impossible for the oxidation silicon vapor-deposited on the surface of the metal, and it has the problem of an evaporation property getting worse remarkably.

[0009] The purpose of this invention has the flat front face of the oxidation silicon vapor-deposited on the base, and moreover, it is to offer the manufacture approach of the oxidation silicon which can be made to exfoliate from a base, without breaking the oxidation silicon.

[0010]

[Means for Solving the Problem] If a metal is used as a base when manufacturing massive oxidation silicon by vacuum evaporation, the oxidation silicon vapor-deposited to the surface of metal will exfoliate from a metal base, without breaking. However, the front face of the obtained oxidation silicon serves as concave convex voice, and becomes EB exposure with an unsuitable thing.

[0011] Considering the front face of the metal base as a **** organization with acid treatment, sandblasting, etc. carried out the knowledge of the effective thing, and this invention person completed this invention, as a result of planning development of the approach of making flat the front face of the oxidation silicon vapor-deposited on the front face of a metal base and continuing experiment research.

[0012] It depends for the karyogenesis consistency in the phase change not only from a gas to oxidation silicon but a solid-state on the boundary tension of the solid-state, molecular weight, a consistency, a degree of supersaturation, temperature, etc. Increase of a degree of supersaturation makes a karyogenesis consistency increase remarkably especially. The karyogenesis consistency was made to increase by the physical approach in this invention, without changing environmental conditions, such as a degree of supersaturation. Because, environmental conditions, such as a degree of supersaturation, have big effect also on a growth step after the birth [from a nucleus]. Since it will have a bad influence on growth of oxidation silicon, it is not desirable to change the conventional environmental condition. The difference in the front face of an unsettled metal and a processing metal is equivalent to the physical effect of above-mentioned. That is, an organization's front face of an unsettled metal is precise, its origination-of-nucleus consistency used as the generation source of solid acid-ized silicon is small at the time of condensation of material gas, and vacuum evaporation, and in order that oxidation silicon may grow locally in a base front face, the front face of the generated oxidation silicon serves as concave convex voice. On the other hand, for the metal base front face which performed surface treatment, it compares with an unsettled surface of metal, and a surface state is rough *****. A karyogenesis consistency increases, local growth is inhibited by this physical factor, and growth takes place to base surface homogeneity according to it. Therefore, the front face of the oxidation silicon to generate will become flat.

[0013] At this time, the karyogenesis rate of oxidation silicon is quick. Because, a karyogenesis rate is influenced by temperature and a rate becomes quick like an elevated temperature. In the vacuum evaporation section, since it reaches enough only with the radiant heat from the lower part beyond karyogenesis need temperature, the karyogenesis rate is quick. If the condition that a karyogenesis rate is quick is not enough as supply of material gas, a nucleus will not grow up to be homogeneity on the whole front face of a metal base, but a part of nuclei will carry out abnormality growth. If it becomes so, the front face of the oxidation silicon generated on the front face of a metal base will become concave

convex voice, and will become EB exposure with an unsuitable thing. It is good to arrange a heating element or a conductor in raw material silicon from this viewpoint. Although its heat absorption is bad since raw material silicon is powder, by arranging a heating element or a conductor in it, and carrying out coincidence heating of the raw material silicon from inside and outside, a vapor rate becomes quick and can make the front face of generation oxidation silicon flatness more.

[0014] It is made easy to exfoliate the oxidation silicon generated by using the metal which considered as the base which carries out evaporation generating of the oxidation silicon, and processed the surface organization to ** from a base front face, and the manufacture approach of the oxidation silicon of this invention carries out as the flat front face for which it was suitable to EB exposure in the oxidation silicon, while preventing that oxidation silicon breaks at the time of the exfoliation.

[0015] Moreover, the front face of oxidation silicon can be made flatter by arranging a heating element or a conductor in raw material silicon.

[0016] As a metal used for a base, the thing excellent in thermal resistance, lightweight nature, and corrosion resistance is required, and can specifically use stainless steel, titanium, etc.

[0017] The processing which makes a metaled front face the organization of ** can use the following approaches. About the chemical technique, there are solvent processing besides [which uses an acid and alkali] a chemical treatment, steam treatment, autoclave processing, etc. In physical means, there are sputter etching processing, shot-blasting processing, tumbling processing, ball mill processing, etc. The contents of the main processing are shown in Table 1.

[0018]

[Table 1]

処 理 名	方 法
酸 処 理	基体を酸（塩酸、沸酸、硝酸、硫酸を単独、あるいは複数混合させて使用する）に浸した後水洗する
アルカリ処理	基体をアルカリ（水酸化カリウム、水酸化ナトリウム、水酸化リチウムを単独、あるいは複数混合させて使用する）水溶液に浸した後水洗する
ボールミル処理	ジルコニアあるいはアルミナ球と共に基体をポットに入れて表面処理を行う
スパッタ処理	基板をターゲットにし、イオンガスとしてArを使用して表面処理する
ショットブラスト処理	鋭角を有する鉄粒を基体表面に噴霧し、表面処理する

[0019] The condition of a surface organization of condensation and rarefaction can be expressed with the magnitude of the surface massive particle for example, by microscope observation, and is desirable. [of the range this / whose / is 50-500 micrometers in this invention] If this is too small, the oxidation silicon which has a flat front face will not generate, but if too large, vacuum evaporation of oxidation silicon will become difficult.

[0020] As a heating element arranged in raw material silicon, a graphite heater can be used like the periphery section. As a conductor, in order to heat raw material silicon, graphite, refractory metals, etc. (a tantalum, tungsten, etc.) which are heated efficient at the heater currently used from the former, and

do not pollute raw material silicon can be used.

[0021]

[Embodiment of the Invention] Drawing 1 explains the gestalt of desirable operation of this invention below.

[0022] The container 2 made from graphite is filled up by using as raw material silicon 1 what mixed silicon powder and quartz powder. A graphite rod is installed in the core of this container 2 as a conductor 3. Apart from this, with ethyl alcohol, acid treatment of the four SUS304 plates as a base 4 is carried out after washing, they are rinsed, and are dried. The base 4 of four sheets is combined in the shape of an rectangular pipe after desiccation.

[0023] The container 2 which was filled up with raw material silicon 1 and installed the conductor 3 is arranged inside the heater 6 in the vacuum chamber 5, and the base 4 combined with the upper part of the vacuum chamber 5 in the shape of an rectangular pipe is attached.

[0024] Evacuation of the inside of the vacuum chamber 5 was carried out, and the raw material silicon 1 in a container 2 was evaporated at the heater 6. The steam adheres to the inside of the base 4 combined in the shape of an rectangular pipe, and deposits oxidation silicon 7.

[0025] At this time, since raw material silicon 1 is fine particles, its thermal conductivity is low. When a conductor 3 does not exist in the interior, it takes most time amount that energy required for evaporation reaches to the core of raw material silicon 1. When a conductor 3 does not exist by installing a conductor 3 in a core, in order for a conductor 3 to absorb the heat which radiated heat in the vertical direction and to transmit the heat to raw material silicon 1 from a core, Raw material silicon 1 will be heated [outside] also from the inside, and the vapor rate of raw material silicon 1 can become quick, and it can make oxidation silicon 7 with a flatter front face generate rather than the time of not installing the conductor 3.

[0026] After checking that oxidization silicon 7 has fully vapor-deposited to the inside of a base 4, the inside of a chamber 5 is cooled, it returns to atmospheric pressure, and a base 4 is taken out.

[0027] The front face of the oxidation silicon 7 vapor-deposited to the inside of a base 4 becomes flat. When making the oxidation silicon 7 exfoliate from a base 4, oxidation silicon 7 does not break. In this way, plate-like oxidation silicon 7 with a flat front face is obtained.

[0028] The obtained plate-like oxidization silicon 7 is cut in the magnitude of arbitration, and it uses for the vacuum deposition ingredient of the plastic film by EB exposure. Since the vacuum evaporatio no ingredient is massive, a splash is not generated, and since it has not broken, the continuous irradiation of EB is possible, and since the front face is still flatter, the homogeneity exposure of EB is also possible. Consequently, the vacuum evaporatio no film of uniform thickness is generated efficiently.

[0029] In order to check the effectiveness of this invention, when it had arranged to the core of a container 2, and acid washing of the base 4 was carried out and a conductor 3 was not installed in the core of a container 2, oxidation silicon 7 was actually manufactured about the case where acid treatment of the base 4 is not carried out. In any case, as shown in Table 2, although oxidation silicon 7 did not break at the time of exfoliation, surface display flatness changed. In addition, Table 2 shows the display flatness of manufacture conditions and Purification SiO.

[0030]

[Table 2]

	製 造 条 件				生成酸化珪素 平坦度*
	酸処理	基体表面塊状 粒子サイズ	発 熱 体 (熱伝導体)	発熱体サイズ	
本発明例	有 り	約 100 μ m	有 り	100 ϕ \times 400 ℓ	1 mm以下
	有 り	約 100 μ m	無 し		1 0 mm以下
比較例	無 し	約10 μ m	有 り	100 ϕ \times 400 ℓ	2 0 mm以下
	無 し	約10 μ m	無 し		2 0 mm以下

* 平坦度 生成酸化珪素断面の最厚部と最薄部の差

[0031]

[Effect of the Invention] Since a metal is used as a base, the manufacture approach of the oxidation silicon of this invention can prevent the oxidation silicon breaking, when making the oxidation silicon vapor-deposited on the base front face exfoliate, as stated above. And since surface irregularity-ization of the oxidation silicon which poses a problem is prevented by roughening processing of a surface of metal when a metal is used, the front face of oxidation silicon can be made flat. Therefore, the massive oxidation silicon suitable for the vacuum evaporation by EB exposure can be manufactured.

[Translation done.]

EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	2	silicon oxide near2 powder same reactor same deposit\$5	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 10:49
L2	78	silicon oxide near2 powder .clm.	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 10:49
L3	53	silicon oxide near2 powder .clm. and react\$6	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 10:56
L4	6	silicon oxide near2 powder .clm. and silicon near2 oxide gas	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 10:57
L5	0	silicon near 2 oxide and "422"/\$.cccls.	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 10:58
L6	0	silicon near 2 oxide and "423"/\$.cccls.	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 10:58
L7	96364	"423"/\$.cccls.	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 10:58
L8	13	7 and silicon near2 oxide powder.clm.	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 11:00
L9	89117	"422"/\$.cccls.	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 11:00
L10	1	9 and silicon near2 oxide powder.clm.	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 11:01
L11	7	9 and silicon near2 oxide powder	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 11:03

EAST Search History

L12	1	9 and silicon near2 oxide gas	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 11:03
L13	15	7 and silicon near2 oxide gas	US-PGPUB; USPAT; EPO; JPO; DERWENT	ADJ	ON	2007/08/19 11:04